

PERFORMANCE CHARACTERIZATIONS OF EURECA RETROREFLECTORS  
WITH FLUOROPOLYMER-FILLED SiO<sub>x</sub> PROTECTIVE COATINGS

Bruce A. Banks  
Sharon K. Rutledge  
NASA Lewis Research Center  
Cleveland, Ohio 44135  
Phone: 216/433-2308, Fax: 216/433-2221

56-27  
1.12  
1.3f

Michael Cales  
Cleveland State University  
Cleveland, Ohio 44115  
Phone: 216/433-2308, Fax: 216/433-2221

#### ABSTRACT

Aluminized corner-cube polymethylmethacrylate retroreflectors were coated with 92% SiO<sub>x</sub>-8% fluoropolymer (by volume) and flown on the EURECA spacecraft. The fluoropolymer-filled SiO<sub>x</sub> protective coating was found to be durable to atomic oxygen when exposed in a ground-based plasma asher to an anticipated mission fluence of  $2 \times 10^{20}$  atoms/cm<sup>2</sup>. Unprotected retroreflector surfaces were found to develop highly diffuse reflectance characteristics, thus inhibiting their use for laser retroreflector purposes. A noncontacting retroreflector optical characterization system was constructed and used to measure the optical retroreflection characteristics of the following retroreflector materials: uncoated unexposed, coated unexposed, both uncoated and coated ground laboratory atomic oxygen exposed and coated exposed to space atomic oxygen exposed and coated exposed to space atomic oxygen on the EURECA spacecraft. A comparison of the results of the optical characterizations is presented.

#### INTRODUCTION

The European Retrievable Carrier (EURECA) Mission included the use of thin, adhesively mounted optical retroreflectors for laser range finding during the retrieval of the EURECA spacecraft. The EURECA spacecraft is a free-flying spacecraft which was launched and retrieved

by the Space Shuttle. The laser retroreflector required durability to atomic oxygen for a fluence in the low  $10^{20}$  atoms/cm<sup>2</sup> range. The acrylic retroreflectors had been used on the Long Duration Exposure Facility (LDEF) and were coated with silicone to prevent optical degradation by atomic oxygen attack (ref. 1, 2). Post-retrieval inspection of the LDEF bicycle reflectors indicated significant degradation in the thin silicone protective coating which had crazed. This allowed atomic oxygen attack of the underlying acrylic corner cube retroreflector. Unprotected polymeric materials are known to be attacked significantly by atomic oxygen producing profusely reflecting surfaces which would not be suitable for laser retroreflector applications (ref. 3, 4). Thus, use of a protective coating applied over the EURECA retroreflector appeared to be a necessity to enable atomic oxygen durability of the specular performance. Ground laboratory and in-space demonstration of SiO<sub>x</sub> and fluoropolymer-filled SiO<sub>x</sub> protective coatings had demonstrated that such protective coatings should be durable to the anticipated EURECA mission fluence (ref. 4, 5). Thus, the application of an SiO<sub>x</sub> or fluoropolymer-filled SiO<sub>x</sub> protective coating over the EURECA retroreflector was evaluated and ultimately used in the first functional application in space of such protective coating systems.

## APPARATUS AND PROCEDURE

The corner cube retroreflectors used for range finding on the EURECA mission consisted of 15.3 cm x 15.3 cm x 0.22 mm thick sheets of aluminized acrylic reflector material with an adhesive backing for attaching to the EURECA spacecraft. A cross section of the retroreflector configuration for both the uncoated and coated configuration ultimately used is shown in Figure 1. Both approximately 1000Å of SiO<sub>x</sub> (where 1.9<x<2.0) and approximately 1000Å of 8% fluoropolymer-filled SiO<sub>x</sub> were evaluated for adhesion to acrylic for retroreflector applications. The fluoropolymer-filled SiO<sub>x</sub> was found to be more spall resistant than pure SiO<sub>x</sub> coatings; and for this reason, was selected for deposition on the EURECA retroreflectors. Deposition of the 8% fluoropolymer-filled SiO<sub>x</sub> coating was accomplished by means of argon ion beam sputter co-deposition from a circular SiO<sub>2</sub> target which had a 5° PTFE Teflon wedge placed on top of it to permit both SiO<sub>x</sub> and fluoropolymer scission fragments to deposit on the retroreflector substrates. Deposition of approximately 1000Å was accomplished in approximately 35 minutes of sputter deposition after initial argon ion precleaning. Sputter precleaning was accomplished by a second ion source (designated as 15 cm ion source) as shown in Figure 2. Descriptions of the improved strain-to-failure properties of such fluoropolymer-filled coatings can be found in references 4 and 6. Samples of EURECA retroreflector material were coated with approximately 1000Å of 8% fluoropolymer-filled SiO<sub>x</sub> and exposed to atomic oxygen along with uncoated laser retroreflector material in a 13.56 MHz RF plasma asher operated on air to a Kapton effective fluence of  $3 \times 10^{20}$  atoms/cm<sup>2</sup>. This exposure was performed to evaluate the optical performance of both the protected and unprotected EURECA retroreflector material to the estimated atomic oxygen mission fluence requirement.

Two coated retroreflectors were then provided for attachment to the EURECA spacecraft scuff plate during the spacecraft integration activities. The EURECA spacecraft was deployed from the Space Shuttle Orbiter cargo bay on August 2, 1992, and retrieved after eleven months in space on June 24, 1993, with a total atomic oxygen fluence of  $2.3 \times 10^{20}$  atoms/cm<sup>2</sup> exposure to the scuff plate surfaces. The scuff plates containing the two retroreflectors were then removed and post-flight optical characterization was performed at Astrotech Corporation on August 3 and 4, 1993. Figure 3 is a photograph of the EURECA spacecraft showing the retroreflectors on the spacecraft as it is being deployed from the Shuttle bay.

Optical characterization of retroreflector materials cannot be reliably accomplished by conventional diffuse or specular reflection measuring techniques because the specular reflection occurs directly parallel to the incoming illumination. This situation frequently prohibits accurate measurement of the return signal. As a result of this complication, it was necessary to construct an optical measurement system which was specifically designed to measure retroreflectance as opposed to conventional specular or diffuse reflectance. Measurement of the retroreflectance was accomplished by using a 670 nm laser and laser power meter in conjunction with a half-silvered mirror as shown in Figure 4. A sand-blasted fused silica window was used as a light diffuser in front of the laser power meter detector to reduce effects of spatial variations in the output of the laser power meter detector. Such spatial variations occur if a narrow beam laser signal arrives at various locations on the laser power meter detector surface. To prevent the laser power meter from viewing reflected illumination, two welders-glass light-trap surfaces were used to absorb the reflected beam by means of multiple reflections and absorptions. A schematic diagram of the optical characterization apparatus is shown in Figure 4. This measurement system was placed in an opaque black cloth enclosure with a hole in it to allow the exit and return of the laser signal, while preventing stray light detection.

## RESULTS AND DISCUSSION

An 8% fluoropolymer-filled SiO<sub>x</sub> ( $1.9 < x < 2.0$ ) protective coating approximately 1000Å thick was found to be durable (based on optical microscope inspections) to atomic oxygen in an RF plasma asher after exposure to a Kapton effective fluence of  $3 \times 10^{20}$  atoms/cm<sup>2</sup>. In contrast, unprotected EURECA retroreflector samples were significantly attacked by atomic oxygen, resulting in a surface with diffusely scattered light, thus greatly attenuating the intensity of retroreflected light. Figure 5 is a photograph of uncoated and 8% fluoropolymer-filled SiO<sub>x</sub> coated EURECA retroreflector materials after exposure to atomic oxygen to a Kapton effective fluence of  $3 \times 10^{20}$  atoms/cm<sup>2</sup> in an RF plasma asher operated on air. The photograph shown in Figure 5 was taken by simultaneously illuminating both samples in a direction parallel to the camera viewing direction. Thus the brightness of the sample, as viewed from the direction of the incident light, is a direct indication of the optical performance of the retroreflectors. The uncoated sample appears dark because it diffusely scattered the incident radiation. The bright protected sample appears bright because it confines its reflected radiation to a specular path

parallel to the incident illumination. However, if one views these two samples from a direction other than parallel to the incident illumination, then the atomic oxygen degraded uncoated sample appears much brighter than the protected sample because of significant scattering of the incident illumination from the rough surface of the unprotected sample.

After exposure in space to an atomic oxygen fluence of  $2.3 \times 10^{20}$  atoms/cm<sup>2</sup> the EURECA laser retroreflectors were fully functional, as can be seen in Figure 6. By comparison of Figures 6a and 6b, one can see that the retroreflectors remained capable of returning a bright specular retroreflection signal. Figure 7 shows a close-up photograph of the retroreflector on the left in Figures 6a and 6b. To the unaided eye, the surface of this retroreflector appeared indistinguishable from samples which were not exposed to the space environment. The right retroreflector appeared identical to the left retroreflector except for a small patch 3 cm in diameter where the protective coating had been damaged by abrasion during pre-flight spacecraft integration activities. Apparently, a bolt adjacent to the scuff plate had to be sawed off, causing repeated contact with the retroreflector in this small area. Although this surface-damaged area was then attacked by atomic oxygen, it provided an ideal opportunity to assess the detrimental effects of atomic oxygen exposure in space to retroreflector material which was not adequately protected.

Optical characterization of the space-exposed retroreflectors required numerous data points to be taken for the various samples at each angle and/or distance because of the great spatial variation in laser retroreflection from the samples. This, in part, is largely due to the fact that the laser beam was approximately 3.5 mm in diameter at 100cm distance, and the corner-cube pattern on the retroreflector material varied in orientation every 4 mm. To average the effects of the spatial variations in retroreflector performance, ten retroreflector measurements were made for each data point averaged and the measurement of the background signal was then subtracted.

Laser retroreflectance characterization of the space-exposed coated EURECA retroreflector materials is shown in Figure 8. This figure compares retroreflectance performance as measured by the optical system shown in Figure 4 for EURECA retroreflector material samples that were coated and space-exposed, coated but not exposed, uncoated and unexposed, coated and plasma asher exposed, uncoated and plasma asher exposed, and the matte abraded spot on the space-exposed retroreflector. Because the standard deviation in retroreflectance for the four high absolute-reflectance surfaces was found to be approximately 6% of their reflectance values, one can conclude that the coated and space-exposed retroreflector had an optical performance indistinguishable from coated samples exposed in a plasma asher. However, unexposed samples which were uncoated or coated had reflectances which were 11 or 17% higher respectively. It is obvious from Figure 8 that unprotected retroreflector materials or protected retroreflector materials whose protection has been abraded away both result in severe loss in retroreflection after exposure to atomic oxygen. These quantified results are also qualitatively witnessed in Figure 5, for the laboratory atomic oxygen environment, and Figure 6b, for the space-exposed atomic oxygen environment results. As can be seen in Figure 6b, the right retroreflector does have a dark spot where the abrasion had damaged the optical performance.

The degradation in retroreflected signal versus distance is shown in Figure 9 for various retroreflector surfaces and exposures. As can be seen from Figure 9, based on the 6% uncertainty in the retroreflection signals, all the retroreflector materials whose coatings or exposures have prevented erosion of the surface of the retroreflector have somewhat similar reduction in return signal with distance. However, the uncoated plasma ashed surface had a much more significant loss of return signal with distance than the surfaces of unexposed or protected retroreflector materials. The more rapid loss of retroreflectance with distance of the uncoated plasma ashed surface is a result of the significant diffuse scattering from microscopically roughened acrylic surface.

The optical performance data illustrated in Figures 8 and 9 were for normal-incident laser radiation. In the realistic environment of space, it is unlikely that laser range finding acquisition will occur at normal incidence to the retroreflectors. Thus, it is important that the retroreflectance signal be high even at off-normal incidence to provide a strong return signal. The range finding was successfully accomplished on EURECA, with the retroreflectors providing a bright visual return signal. Figure 10 compares the retroreflectance relative to normal incidence as a function of angle of illumination relative to the surface normal for the various retroreflector surfaces. As can be seen from Figure 10, the EURECA coated and space-exposed retroreflectors had an angular dependence which was indistinguishable from pristine retroreflector surfaces or ones which were coated and exposed to a simulated low Earth orbital environment using RF asher plasma atomic oxygen.

Based on the astronauts' satisfaction with the performance of the retroreflectors, as well as the quantified optical performance shown in Figures 8, 9, and 10, one can conclude that atomic oxygen protection is needed and that 8% fluoropolymer-filled  $\text{SiO}_x$  protective coatings provide excellent atomic oxygen protection of acrylic retroreflector materials for atomic oxygen fluences up to  $2.3 \times 10^{20}$  atoms/cm<sup>2</sup>.

## SUMMARY

Unprotected acrylic laser retroreflective materials were found to be highly degraded by atomic oxygen produced by ground laboratory RF plasma ashers. Similar optical degradation results were observed for atomic exposure in space on a small portion of a protected EURECA retroreflector which had its protection removed by abrasive damage prior to launch. Thus, it appears that acrylic laser retroreflectors require functional coatings to provide acceptable retroreflectance signals after exposure in low Earth orbit.

A 1000Å sputtered coating of 8% fluoropolymer-filled  $\text{SiO}_x$  ( $1.9 < x < 2.0$ ) was found to provide excellent protection of laser retroreflector materials both in ground-based plasma asher tests and in space on the EURECA spacecraft which was exposed to an atomic oxygen fluence of  $2.3 \times 10^{20}$  atoms/cm<sup>2</sup>. The optical performance of the protected EURECA retroreflectors was found to be

indistinguishable from protected retroreflectors which were exposed to atomic oxygen in an RF plasma asher. The dependence of retroreflectance on distance and angle of arriving illumination was similar for both in-space and ground laboratory atomic oxygen exposed retroreflector samples.

#### REFERENCES

1. Banks, B. A., Dever, J. A., Gebauer, L., and Hill, C. M.: Atomic Oxygen Interactions with FEP Teflon and Silicones on LDEF. Proceedings of the First LDEF Post-Retrieval Symposium, Kissimmee, FL, June 2-8, 1991.
2. Banks, B. A., Rutledge, S. K., de Groh, K. K., Mirtich, M. J., Gebauer, L., Olle, R., and Hill, C. M.: The Implications of the LDEF Results on Space Station Freedom Power System Materials. Proceedings of the Fifth International Conference on Materials in a Space Environment, Cannes, France, September 16-20, 1991.
3. Lejer, L. J.: Oxygen Atom Reaction with Shuttle Materials at Orbital Altitudes. NASA TM-58246, 1982.
4. Banks, B. A., Mirtich, M. J., Rutledge, S. K., Swec, D. M., and Nahra, H. K.: Ion Beam Sputter-Deposited Thin Film Coatings for Protection of Spacecraft Polymers in Low Earth Orbit. Proceedings of the 23rd Aerospace Sciences Meeting, Sponsored by the American Institute of Aeronautics and Astronautics, Reno, NV, January 14-17, 1985.
5. Rutledge, S. K., and Olle, R. M.: Durability Evaluation of Photovoltaic Blanket Materials Exposed on LDEF Tray S1003. Proceedings of the First LDEF Post-Retrieval Symposium, Kissimmee, FL, June 2-8, 1991.
6. Banks, B. A., Rutledge, S. K., de Groh, K. K., LaMoreaux, C., and Olle, R.: Fluoropolymer Filled SiO<sub>2</sub> Coatings; Properties and Potential Applications. Proceedings of the Society of Vacuum Coaters 35th Annual Technical Conference, Baltimore, MD, March 22-27, 1992.

FIGURES

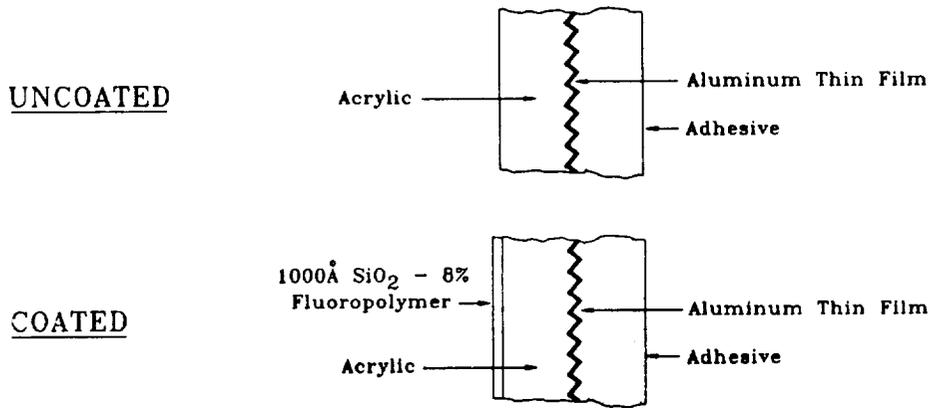


Figure 1. Cross section of uncoated and coated EURECA retroreflector material.

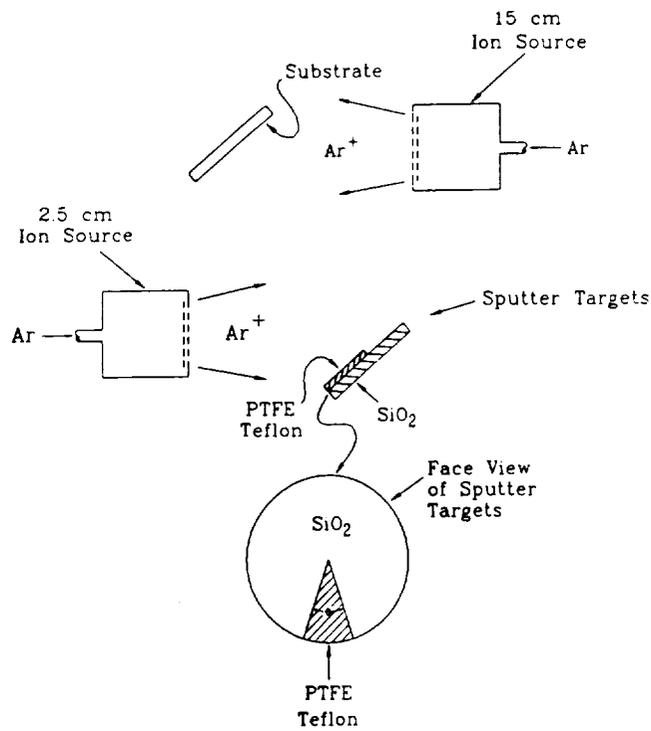


Figure 2. Ion beam sputter coat deposition process for deposition of fluoropolymer-filled SiO<sub>x</sub> protective coatings.

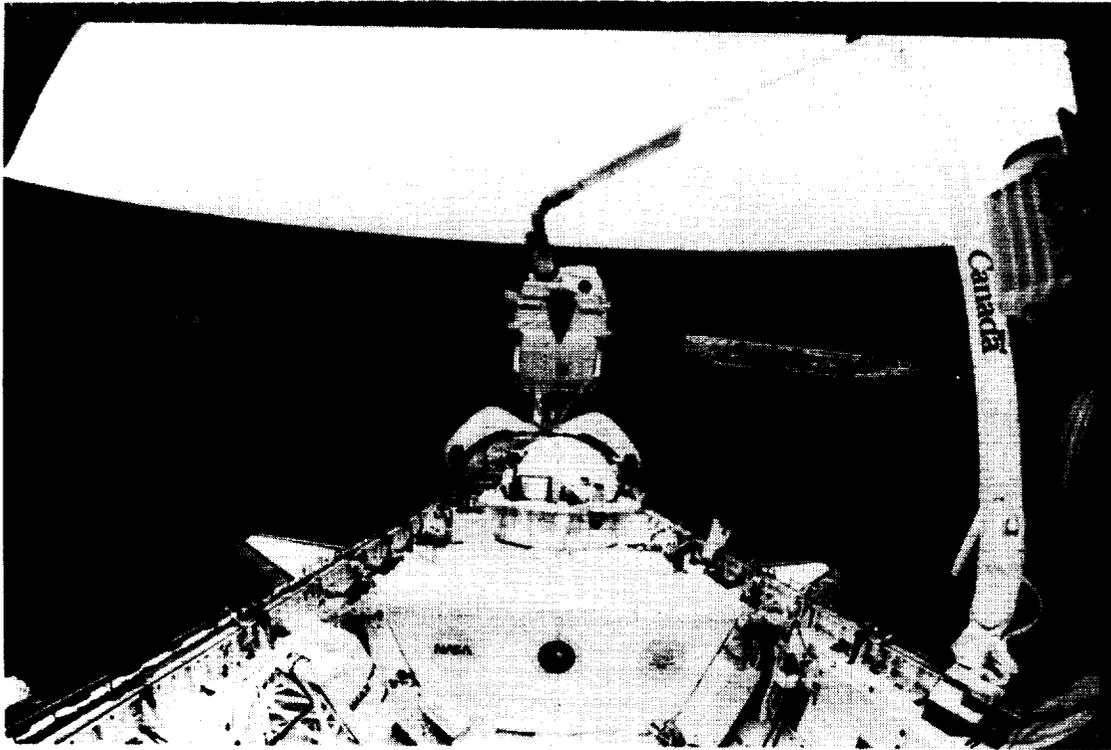


Figure 3. Photograph showing the two laser retroreflectors on the EURECA spacecraft as it is being deployed from the cargo bay of the Space Shuttle on August 2, 1992.

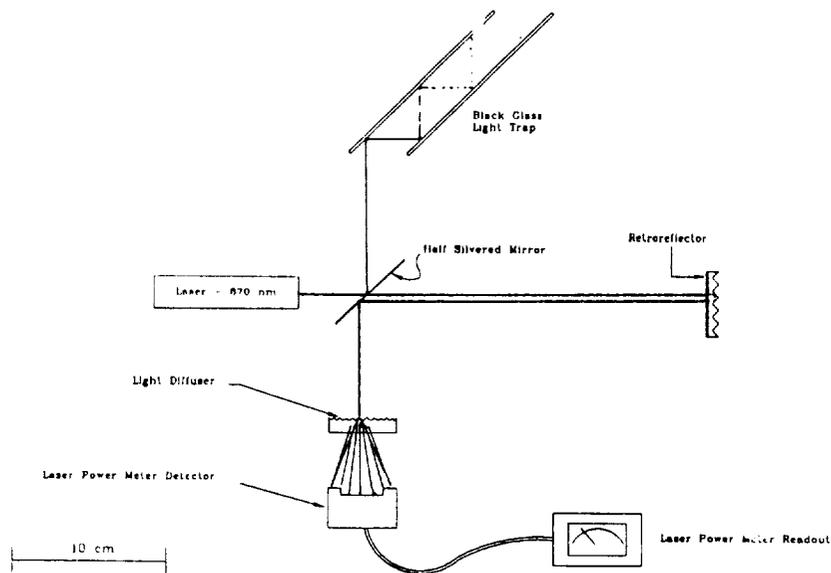


Figure 4. Optical system used to measure the retroreflectance of the EURECA retroreflectors.

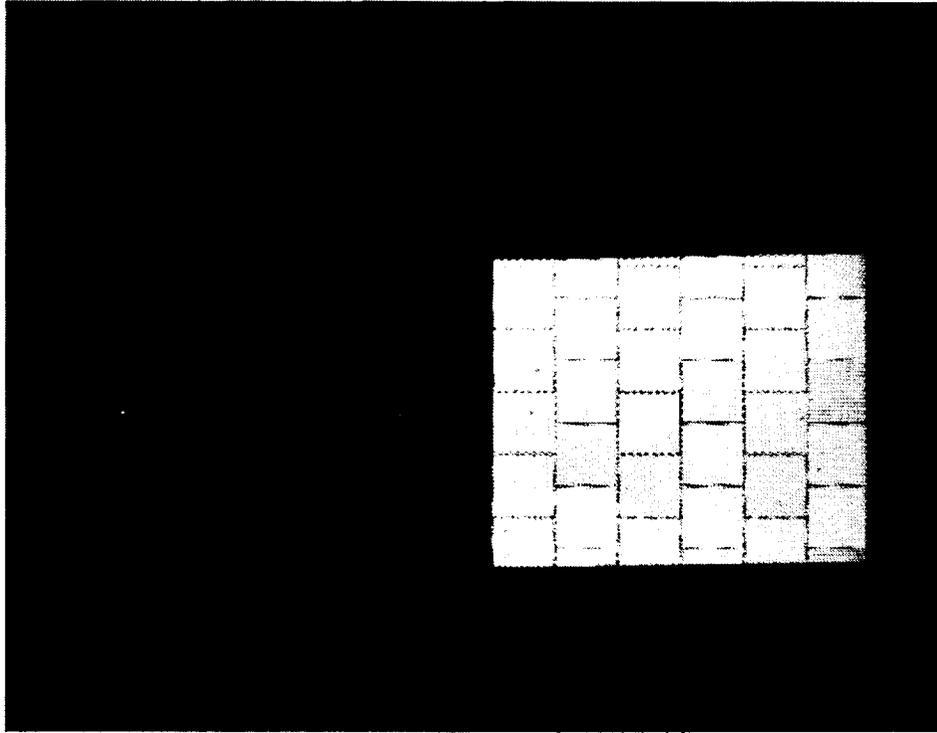


Figure 5. Photograph of uncoated (dark image on the left) and approximately 1000Å thick 8% fluoropolymer-filled SiO<sub>x</sub> coated (bright image on the right) EURECA retroreflector samples after exposure to a Kapton effective atomic oxygen fluence of  $3 \times 10^{20}$  atoms/cm<sup>2</sup>.

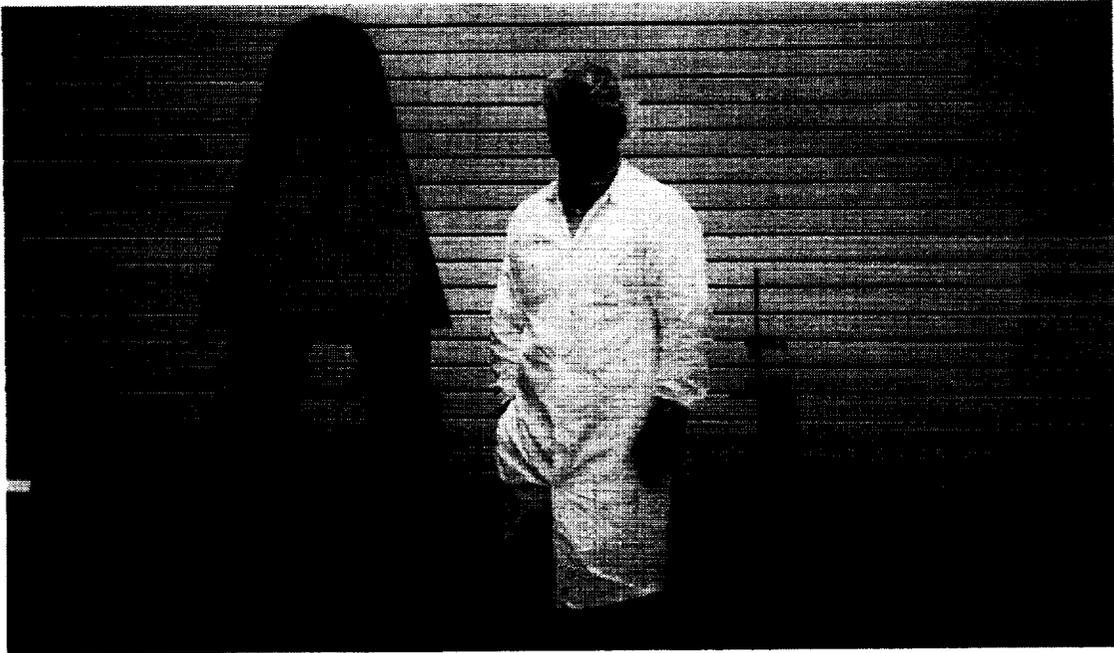


Figure 6a. Photographed with ceiling illumination only.

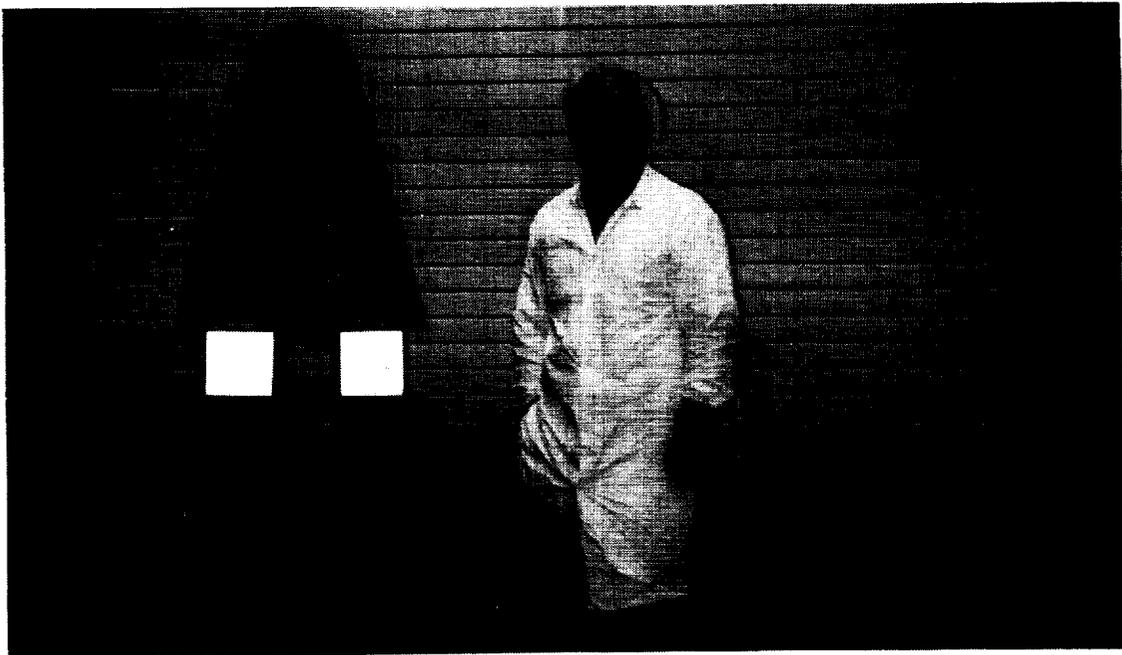


Figure 6b. Photographed with ceiling illumination and incandescent lamp illumination parallel to the camera viewing direction.

Figure 6. Fluoropolymer-filled  $\text{SiO}_x$  protected EURECA laser reflectors mounted on the EURECA scuff plate after retrieval from in-space exposure.

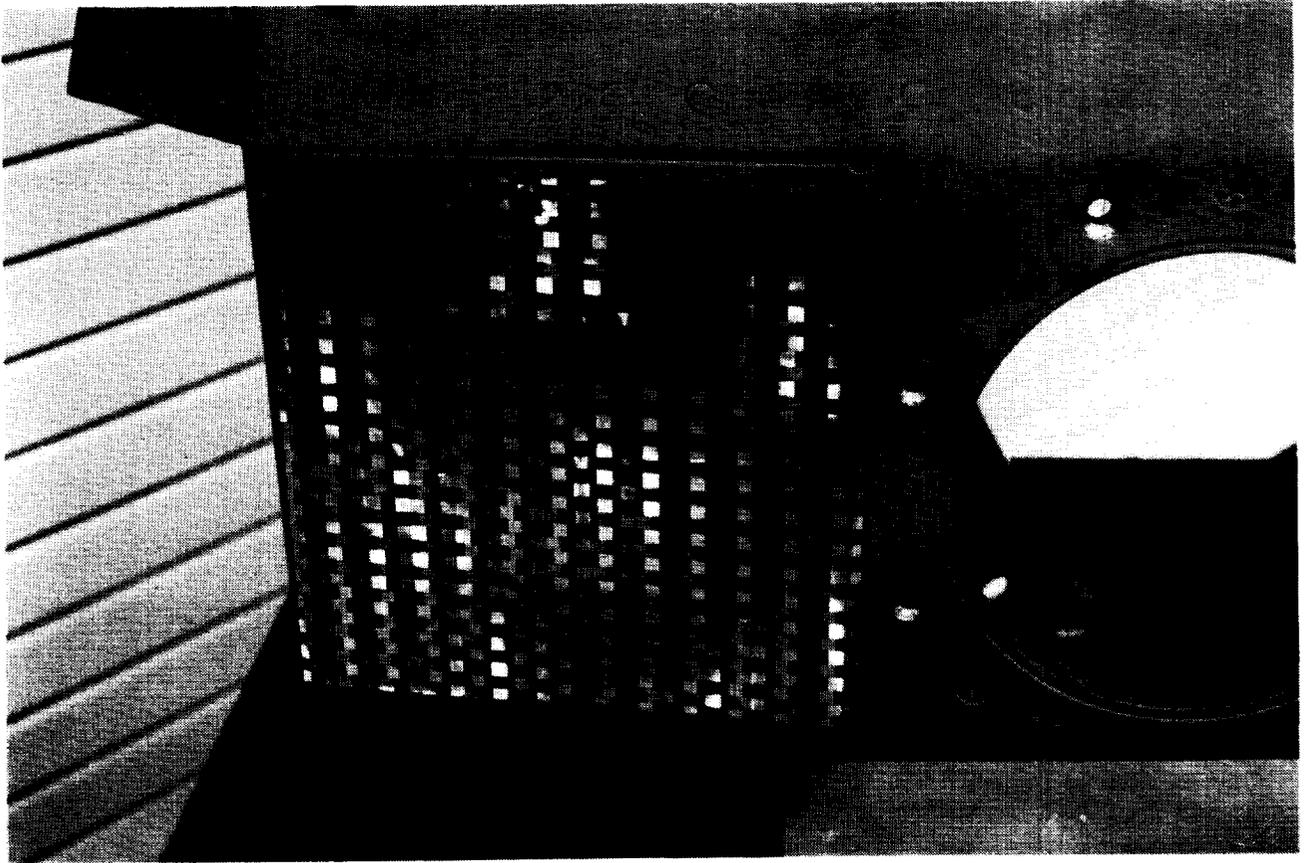


Figure 7. Close-up photograph of left EURECA retroreflector after retrieval from space.

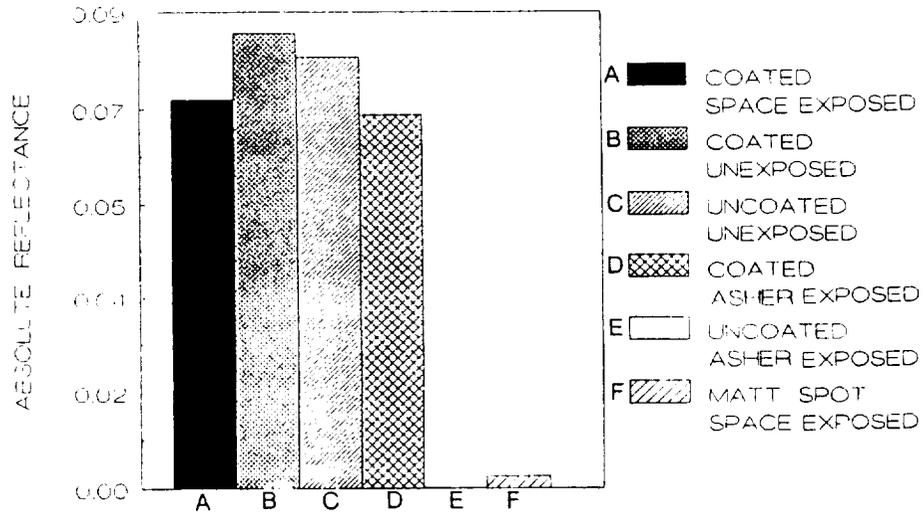


Figure 8. Comparison of optical performance of EURECA retroreflector materials for 670 nm illumination.

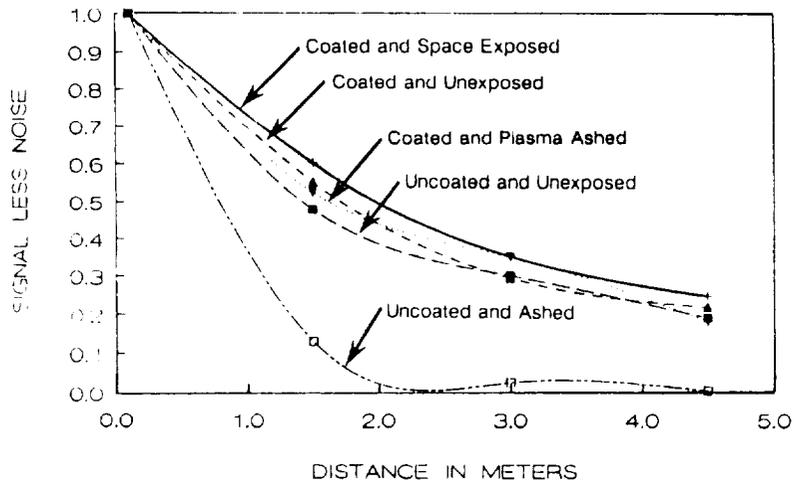


Figure 9. Retroreflector optical performance for coated and space exposed, coated and unexposed, coated and plasma ashed, uncoated and unexposed, and uncoated and plasma ashed retroreflector materials illuminated with 670 nm wavelength light.

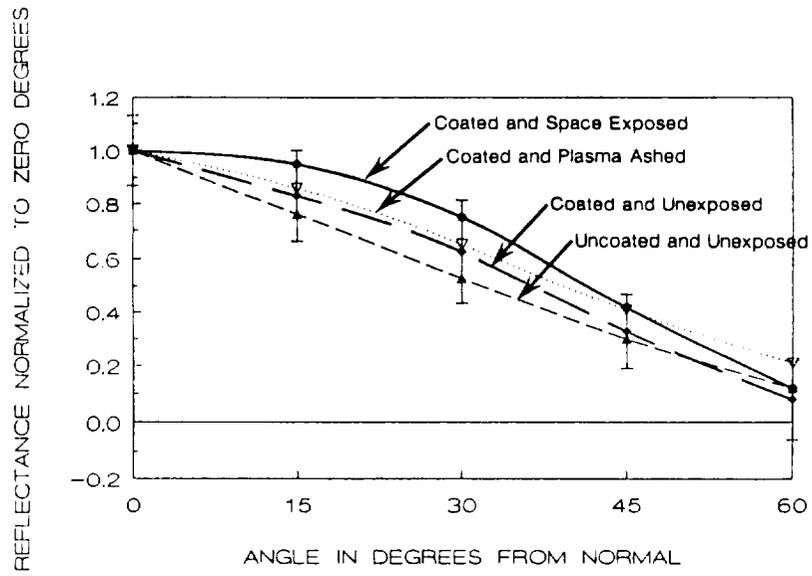


Figure 10. Angular dependence of retroreflectance for coated and space exposed, coated and plasma ashed, coated and unexposed, uncoated and unexposed retroreflector materials illuminated with 670 nm wavelength light.

